An experimental investigation on nano-TiO_2 and fly ash based high performance concrete

Tiago Martins, F. Pacheco Torgal, Sérgio Miraldo, José Barroso Aguiar and Jesus Carlos

High performance concrete (HPC) offers several advantages over normal-strength concrete, namely, high mechanical strength and high durability. Therefore, HPC allows for concrete structures with less steel reinforcement and a longer service life, both of which are crucial issues in the eco-efficiency of construction materials. Nevertheless international publications on the field of concrete containing nanoparticles are scarce when compared to Portland cement concrete (around 1%) of the total international publications. HPC nanoparticle-based publications are even scarcer. This article presents the results of an experimental investigation on the mechanical properties and durability of HPC based on nano-TiO_2 and fly ash. The durability performance was assessed by means of water absorption by immersion, water absorption by capillarity, ultrasonic pulse velocity, electric resistivity, chloride diffusion and resistance to sulphuric acid attack. The results show that the concretes containing an increased content of nano-TiO_2 show decreased durability performance. The results also show that concrete with 1% nano-TiO_2 and 30% fly ash as Portland cement replacement show a high mechanical strength (C55/C67) and a high durability. However, it should be noted that the cost of nano-TiO_2 is responsible for a severe increase in the cost of concrete mixtures.

Keywords: *Portland cement; fly-ash; TiO_2 nanoparticles; compressive strength; durability; HPC; cost*

1. INTRODUCTION

The expression 'high-performance concrete' was coined by Professor Roger Lacroix and Professor Pierre-Claude Aïtcin in 1980 [1]. However, only a small part of current concrete production falls on this category. According to the ERMCO statistics [2], ready-mixed concrete strength class production lies essentially between C25/30 and C30/37. Additionally, only 11% of the concrete production corresponds to the HPC strength class target. It is worth noticing that, according to Hegger et al. [3], the increase of compressive strength in concrete imply a reduction in reinforced steel amount by as much as 50%. Normal-strength class concrete means less durable concrete structures which, in turn, require frequent maintenance and conservation operations or even the structure´s entire replacement (associated with the consumption of additional raw materials and energy). Besides many of the degraded concrete structures were built decades ago, when little attention was given to durability issues [4]. It is no surprise then, that worldwide concrete infrastructure rehabilitation costs are staggering. In fact, in the USA about 27% of all highway bridges are in need of repair or replacement. Plus, the corrosion deterioration cost due to deicing and sea salt effects is estimated at over 150 billion dollars [5]. Beyond the durability problems originated by imperfect concrete placement and curing operations, the real issue of ordinary Portland cement concrete (OPC) durability is related to the intrinsic properties of that material. In effect, it presents high permeability which, in turn, allows water and other aggressive elements to enter, leading to carbonation and chloride ion attack ultimately resulting in corrosion problems [6, 7]. The importance of durability, in the context of construction and building materials eco-efficiency has been rightly put by Mora [8]. This author stated that increasing concrete durability from 50 to 500 years would mean a reduction of its environmental impact by a factor

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of 10. Nanotechnology involves the study at microscopic scale (1 nm = 1×10^{-9} m). The use of nanoparticles to increase the strength and durability of cementitious composites was already predicted by the report RILEM TC 197-NCM, "Nanotechnology in construction materials", to be a research area with high nanotech potential [9]. Nevertheless international publications on the field of concrete with nanoparticles are scarce when compared to concrete total international publications (around 1%). HPC nanoparticle based publications are even scarcer [10]. Portland cement replacement by some supplementary cementitious material, like fly ash can contribute to a more eco-efficient concrete production. However, fly ash has very slow hydration characteristics thus providing very little contribution to early age strength [11]. Partial replacement of Portland cement by 30% fly ash leads to a relevant early decrease in compressive strength as much as 40% at 28 days curing [12]. This is why European standard EN 197 limits the Portland cement replacement ratio to under 35% for type II cements [13]. Since nanoparticles have a high surface area to volume ratio providing high chemical high reactivity they could be used to overcome the limitations of fly ash incorporation. Thus meaning that investigations on the mechanical properties and durability of HPC based on nano-TiO_2 and fly ash are needed.

2. EXPERIMENTAL WORK

2.1 Materials, mix design and concrete mixing

The characteristics of the aggregates used to make the concrete mixtures are shown in Table 1 and in Figure 1.

An ordinary Portland cement (CEM I 42,5) was used. The fly ash was supplied by and according to the NP EN 450-1 it belongs to B class and has an N class fineness modulus. A second generation superplasticizer (SP) based on polycarboxylic ether polymers was used at appropriate percentages in order to retain the slump of the fresh concrete between 100 and 150 mm (class S3 of NP EN 206-1 [14]). In order to find the most suitable content of SP several cement pastes with a w/c=0.3 were tested with the Marsh cone using several SP contents (1%, 1.5%, 1.7%, 2%, 2.5% and 3%). Figure 2 shows that the mixture with 2% SP is the most suitable providing the less flow time for the less SP content.

Table 1. Characteristics of the aggregates

	Max dimension	Fine content	Density (kg/m^3)	Water absorption
Sand	4.0	≤3	2660	0.2
Coarse aggregates	8.0	≤1.5	2620	0.6

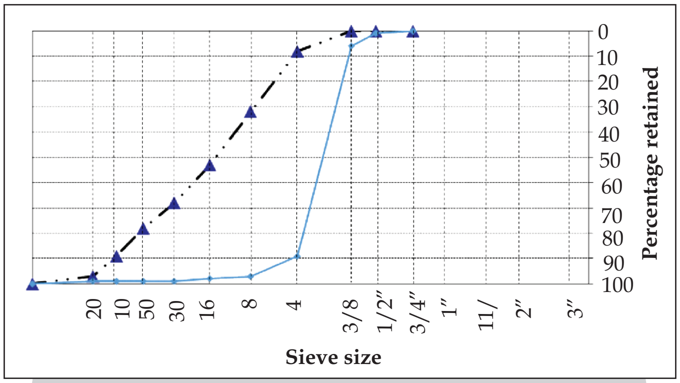

Figure 1. Aggregate particle size distribution of the sand and of the coarse aggregate

Commercially available nano-TiO_2 powder from a reputed company was used in three different contents (1%, 2% and 3%) by cement mass. The particle size of the TiO_2 is 21 nm, with a specific BET surface area of 50 m^2/g. Although the use of nanoparticles is very recent, it has already raised issues concerning its potential toxicity. Some investigations showed that nanoparticles can cause symptoms like the ones caused by asbestos fibres [15]. Therefore, during the mortar mixing masks and gloves were used to avoid contact with the nano-TiO_2 powder. The nano-TiO_2 powder was previously mixed with Portland cement during 5 minutes in order to increase its dispersion. Several concrete mixes with a water/binder ratio of 0.35 and 500 kg/m^3 of binder were designed using the Faury concrete mix design method. In a first set three mixtures with increasing nano-TiO_2 contents (1%, 2% and

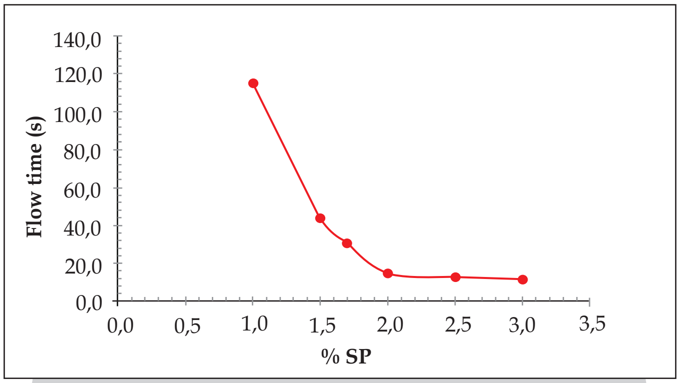

Figure 2. Fluidity curve

3%) were evaluated for compressive strength. Then the mixture with the nano-TiO_2 content that led to the highest compressive strength was chosen to be used in the mixture with partial replacement of Portland cement by 30% fly ash. The six concrete mixes are described in Table 2.

Table 2. Concrete mix proportions per cubic meter of concrete

	Cement (kg)	Fly ash (kg)	TiO_2 kg	Sand (kg)	Coarse aggregates (kg)	Water (l)	SP (l)
C_ref	500	-	-	852	823	182	10.0
C_1TiO2	496	-	4.1	765	848	182	10.0
C_2TiO2	491	-	9.4	601	889	182	10.0
C_3TiO2	484	-	15.6	453	915	182	10.0
C_30CV	350	150	-	809	852	169	10.0
C_1TiO2_30CV	345	150	4.4	698	882	169	10.0

Table 3 shows the cost of materials which were used for the calculation of the percentage variation of concrete mixtures related to the reference mixture (Figure 3). The results show that the use of nanoparticles even as low as just 1% leads to a severe increase in the concrete cost. Even the replacement of 30% of Portland cement by fly ash only allows for a reduction from 118% to 112%. This means that the economic feasibility of nano-TiO_2 based concrete is dependent on the reduction of the cost of nanoparticles. This cost considerations does not even include any cost increase due to the use of safety measures related to the handling of nanoparticles.

3 EXPERIMENTAL PROCEDURES

3.1 Compressive strength

The compressive strength test was performed under NP EN 206-1[14]. The concrete specimens were conditioned at a temperature equal to 21 ± 2 ºC cured in a moist chamber until they had reached the testing ages. Tests were performed on 150x150x150 mm^3 specimens. The compressive strength for each mixture was obtained from an average of 3 cubic specimens and determined at the age of 7 and 28 days of curing.

3.2 Water absorption by immersion

Tests were performed on 100x100x100 mm^3 specimens. The specimens were tested at 28 days of curing. The specimens were immersed in water at room temperature for 24 hours. First the weight of the specimens while suspended by a thin wire and completely submerged in water is recorded as Wim (immersed weight). After that, the specimens were removed from water and placed for 1 min on a wire mesh. This allows water to drain. Then, visible surface water is removed with a damp cloth and weight is recorded as Wsat (saturated weight). All specimens were placed in a ventilated oven at 105 ºC for not less than 24 hours while allowing for two successive weightings at intervals of 2 hours to show an increment of loss not greater than 0,1% of the last previously determined weight of the specimen. The weight of the dried specimens is recorded as Wdry (oven-dry weight). Absorption coefficient is determined as following equation :

$$A(\%) = \frac{W_{sat} - W_{dry}}{W_{sat} - W_{im}} \times 100 \qquad ...(1)$$

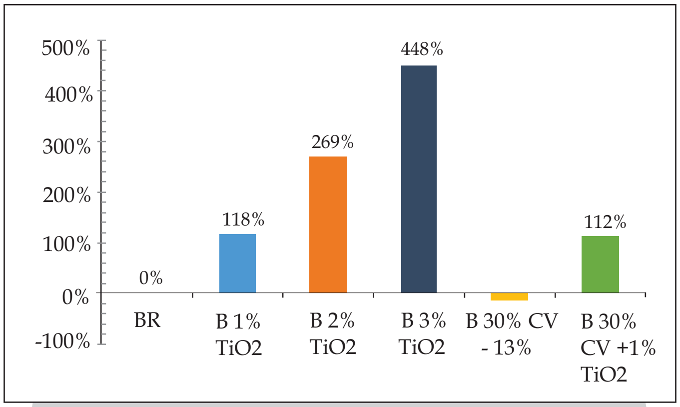

Figure 3. Variation on the percentage cost of concrete mixtures

3.3 Capillary water absorption

The capillary water absorption was assessed using cubic specimens 10 cm high. After 28 days in a moist chamber the specimens were placed in an oven 45 ºC for 14 days. The test consists in placing the specimens in a container with enough water to maintain immersed one of the sides of the sample. This test is carried on according to Standard LNEC E393 [16]. Water absorption was measured after (5, 10, 20, 30, 60, 90, 120, 180, 240, 300, 360, 420, 480) minutes. Capillarity water absorption was obtained from an average of 3 specimens.

Table 3. Cost of materials [in euro/kg (and in Indian rupee/kg)]

Portland Cement	Fly ash	Nano TiO_2	Sand	Coarse aggregates	Water	SP
0.1 (6.99)	0.03 (2.10)	25.6 (1789.6)	0.008 (0.56)	0.007 (0.49)	0.1 (6.99)	0.82 (57.32)

3.4 Ultrasonic pulse velocity

The ultrasonic pulse velocity test was performed was performed under the NP EN 12504-4 [17] and using 100x100x100 mm^3 specimens. Readings were recorded for 7 and 28 days of curing. The ultrasonic velocity was measured by direct way, through the cylindrical specimen and between the two parallel sides.

3.5 Electric resistivity

Electric resistivity was obtained from an average of 3 cylinders with 100 mm diameter and 200 mm high. The electric resistivity of the concrete specimens were performed using the four-point Werner electrode according to others [18]. Prior to measurements, the specimen's surfaces were cleared of excess water with a dry cloth. The specimens were measured on three lateral sides, two readings on each side with a 180^o rotation (six readings per specimen). Readings were performed for 7 and 28 days of curing. Corrosion risk was assessed through the recommendation of the European Concrete Committee (CEB 192) which is represented in Table 4.

3.6 Chloride diffusion test

This test method consists of the determination of the depth of penetration of chloride ions through 50 mm thick slices of 100 mm nominal diameter cylinders. This test his performed in according to LNEC E 463 [19]. A potential difference of 30±0,2V is maintained across the specimen. One face is immersed in a sodium chloride and sodium hydroxide solution, the other in a sodium hydroxide solution. The chloride diffusion coefficient can be calculated using the following equation:

$$D = (RTL/zFU).[X_d - (\alpha \sqrt{X_d})/t]$$

where:
$\alpha = 2\sqrt{(RTL/zFU)}.\ erf^{-1}(1-2c_d/c_o)$
D = diffusion coefficient, m2/s;
z = absolute valence of the ion involved, for chloride ion, z = 1;
F = Faraday constant, F = 9.648 x 104 J/(V.mol);
U = absolute potential difference, V;
R = constant of ideal gases, R = 8.314 J/(K.mol);
T = solution temperature, K;
L = thickness of specimen, m;
X_d = depth of penetration, m;
t = duration of the test, seconds;
erf^{-1} = inverse of error function;
c_d = chloride ion concentration with which the colour changes;
c_o = concentration of chloride ion in the sodium chloride solution.

Table 4. Corrosion risk according to concrete resistivity (CEB 192)

Concrete resistivity (Ω.m)	Corrosion risk
<50	Very high
50-100	High
100-200	Low
>200	Very low

3.7 Resistance to sulphuric acid attack

The resistance to acid attack followed a variation of the ASTM C-267 (Standard test methods for chemical resistance of mortars, grouts, and monolithic surfacings and polymer concretes).

The test used in the present investigation consists in the immersion of 100×100×100 mm^3 concrete specimens with 28 days curing in a 10% of sulphuric solution during 28 days. The resistance to acid attack was assessed by the differences in weight of dry specimens before and after acid attack at 1, 7, 14, 28 and 56 days.

4. RESULTS AND DISCUSSION

4.1 Compressive strength

Figure 4 shows the compressive strength of the six concrete mixtures at 7 days and 28 days. The results show that mixtures with partial replacement of Portland cement by 1% nano-TiO_2 have the same compressive strength as the reference mixture. Both for 7 and 28 days curing days. This could be filler effect related. The increase in the nano-TiO_2 content leads to a decrease in the compressive strength

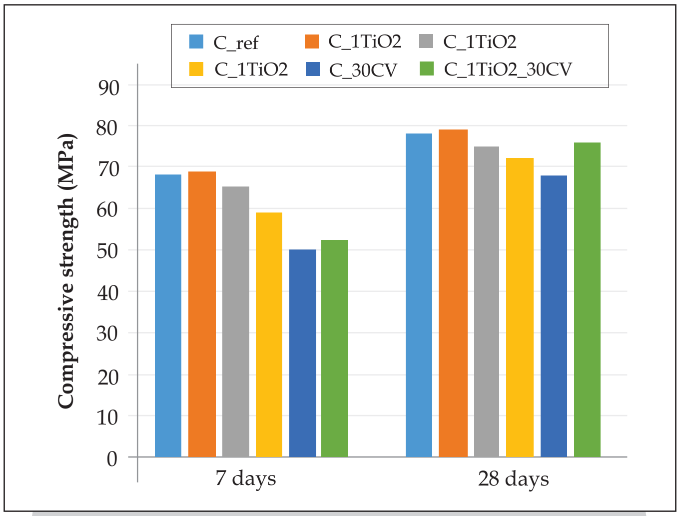

Figure 4. Compressive strength according to curing days

both for 7 and 28 days curing days. Still the compressive strength decrease is worse in the early ages. This could be due to unsuitable dispersion of nanoparticles in the concrete matrix as per previous investigations [20]. A 1% nano-TiO_2 content seems to be an optimal percentage as already found by others [21-23]. When compared to the reference mixture, the 3% nano-TiO_2 mixture has a 13% compressive strength decrease after 7 days curing but only an 8% decrease after 28 days curing. Meaning that the hydration is accelerated by the nano-TiO_2 presence. The concrete mixture with mixture with partial replacement of Portland cement by 30% fly ash shows an almost 30% compressive strength decrease after 7 days curing confirming that fly ash provides very little contribution to early age strength. This is related to the reactivity of the fly ash used in this investigation. If a class C fly ash with high fineness and high reactivity would have been used higher compressive strength results could have been achieved. The results show that the nano-TiO_2 minimizes the strength loss associated to the use of fly ash but only for 28 days curing. At early ages the contribution of nano-TiO_2 for the compressive strength of the fly ash mixture is null.

4.2 Water absorption by immersion

The water absorption results of the six concrete mixtures are shown in Figure 5. The results show that for mixtures without fly ash increasing nano-TiO_2 content leads to an increase in water absorption. A valid statistical correlation (R^2=0.98) was obtained between water absorption and 28 days curing compressive strength for the mixtures without fly ash (Figure 6). Previous investigations [24] seem to confirm this behavior stating that the increasing content of nano-particles leads to a coarser pore structure of concrete. The mixture with partial replacement of Portland cement by 30% fly ash shows an almost 20% water absorption reduction. Part of

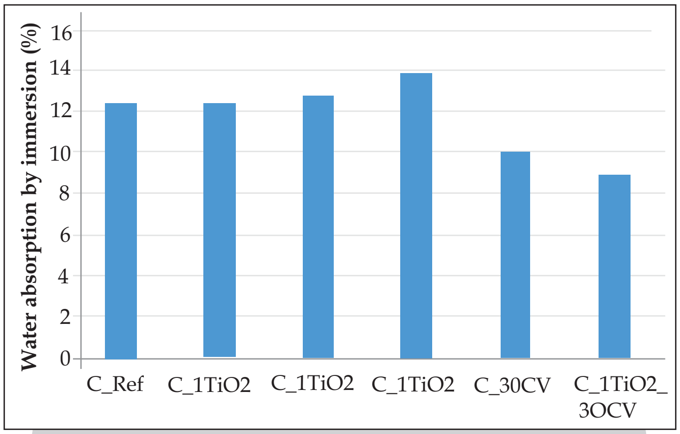

Figure 5. Water absorption by immersion

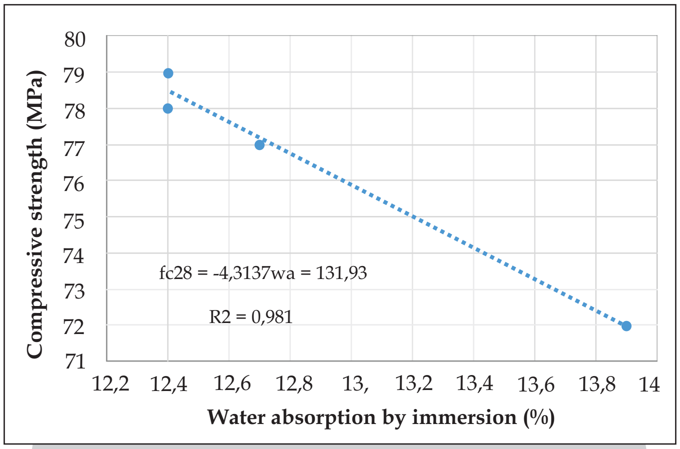

Figure 6. Statistical correlation between 28 days curing compressive strength and 28 days curing water absorption by immersion

the explanation lies in the 7% difference between the w/b of the two mixtures. The remaining 23% gap could be related to the fact that since the pozzolanic reaction has consumed $Ca(OH)_2$ while generating more CSH, this could lead to a denser microstructure. The mixture with 30% fly ash and 1% nano-TiO_2 shows an even lesser water absorption, reflecting the acceleration of the pozzolanic reaction.

4.2 Capillary water absorption

Figure 7 shows the capillary water absorption coefficients. Concerning the mixtures without fly ash the results reveal that nano-TiO_2 content is associated to an increase in capillary water absorption. This follows the same trend already observed for water absorption by immersion. Meaning that a 3% nano-TiO_2 content leads to a high internal capillary microstructure. Other authors [25] studied similar concrete compositions (without nano-TiO_2) with similar w/b reported that the mixture with 30% fly ash had much higher capillary

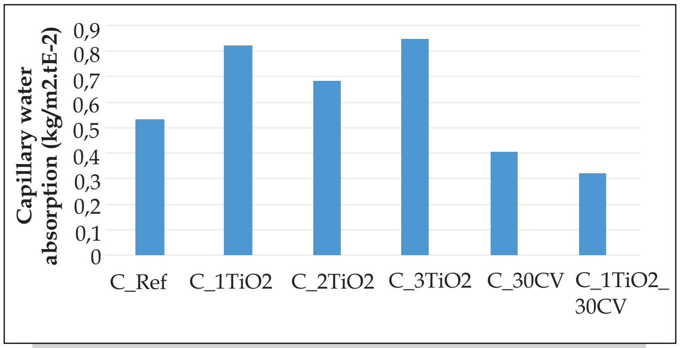

Figure 7. Capillary water absorption coefficients

than the reference mixture. The only differences between them concerns the type II cement (clinker content between 65% and 79%) in previous investigations and type I (clinker content between Capillary water absorption (kg/m2.tE-2) 95% and 100%) in the current study.

4.3 Ultrasonic pulse velocity (UPV)

The ultrasonic pulse velocities are shown in Figure 8. The UPV at 7 days curing is lower than for 28 days curing for all the six concrete mixtures. A similar behavior was observed for the compressive strength meaning that a denser microstructure is associated with high ultrasonic pulse velocities. Figure 9 shows that a significant statistical correlation (R^2=0.98) was obtained between UPS and compressive strength both for 7 days curing and 28 days curing.

4.4 Electric resistivity

Figure 10 shows the electric resistivity. At 7 days curing all mixtures show a lower electric resistivity than the reference mixture. Still, according to the CEB 192 (Table 4) the corrosion risk, for all the 7th curing days mixtures falls below the very high range. For 28 days curing the mixtures with fly ash outperform the reference mixture. At this curing age all the mixtures have low corrosion risk. The mixture with fly ash and 1% nano-TiO_2 content shows very low corrosion risk. Since electrical resistivity is one of the main parameters controlling the initiation and propagation of reinforcement corrosion [26], the use of 30% fly ash and 1% nano-TiO_2 content based concrete seems to be a very effective option.

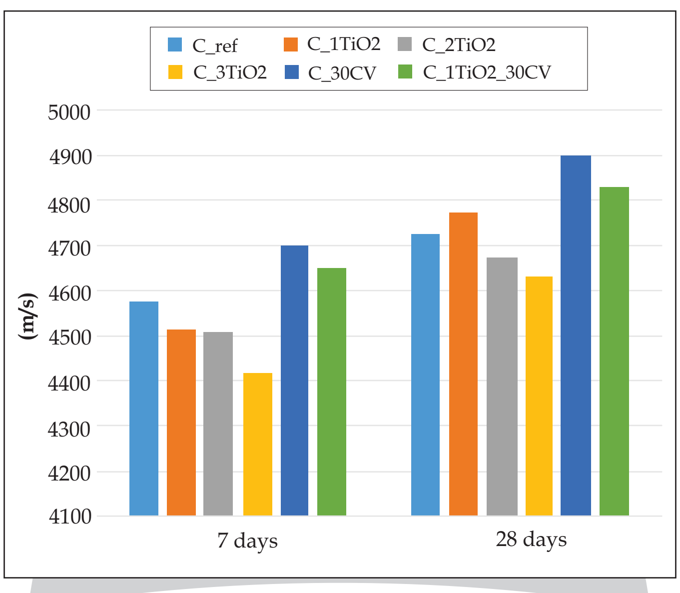

Figure 8. Ultrasonic pulse velocity

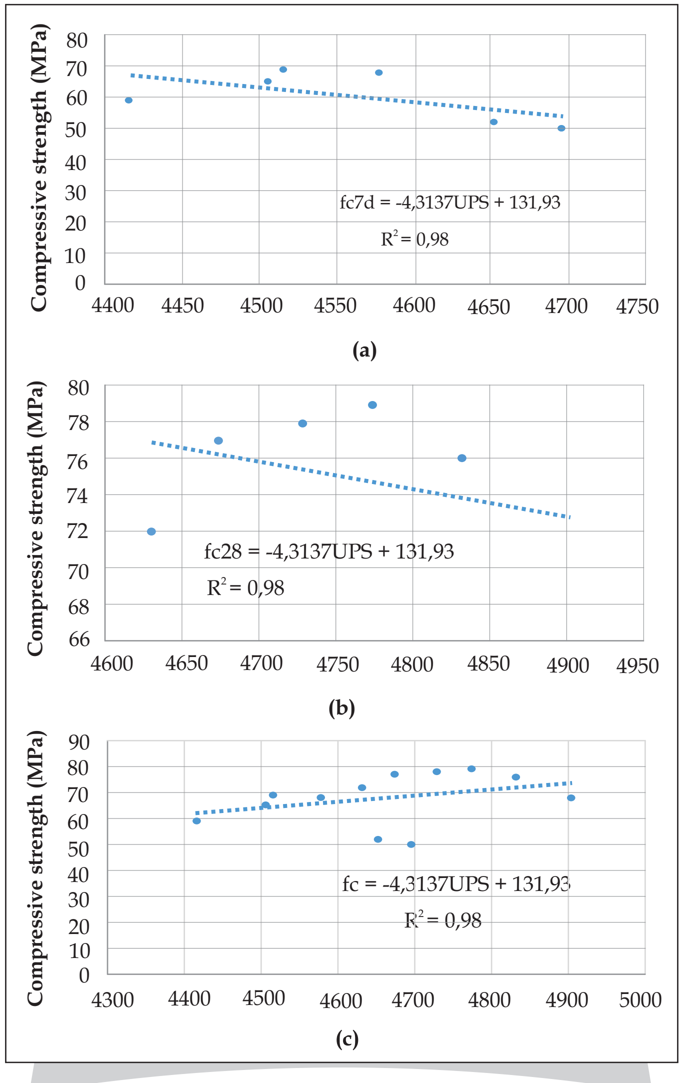

Figure 9. Statistical correlation between compressive strength and UPS: a) For 7 days curing; b) For 28 days curing; c) For all mixtures both at 7 and 28 days curing

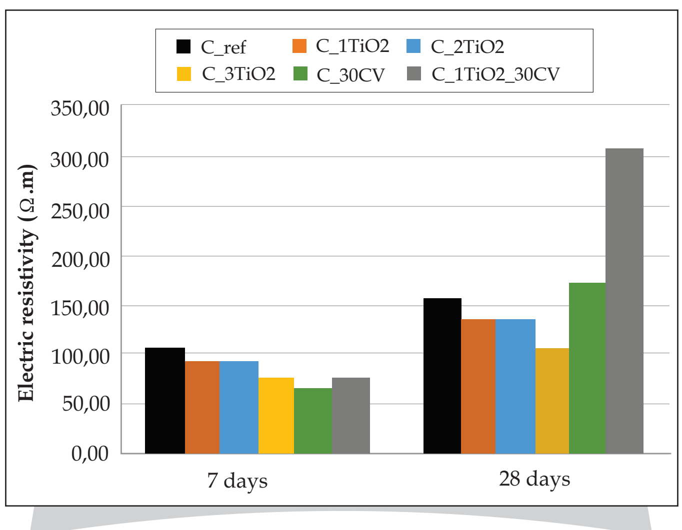

Figure 10. Electric resistivity

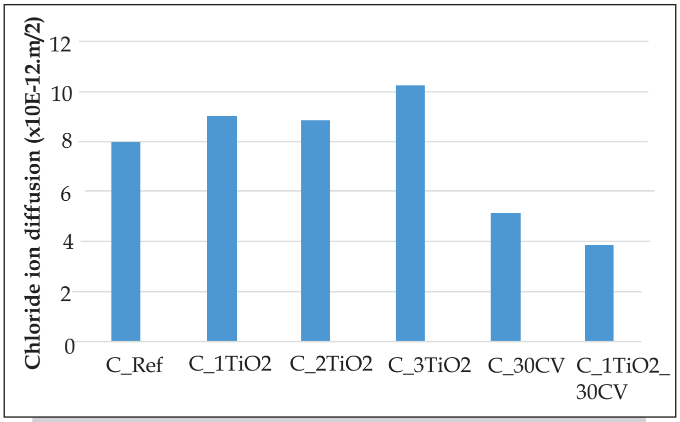

Figure 11. Chloride ion diffusion coefficient

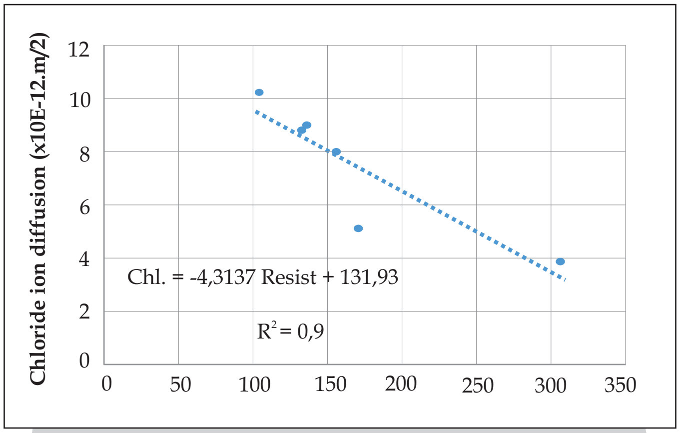

Figure 12. Statistical correlation between chloride ion diffusion coefficient and electric resistivity

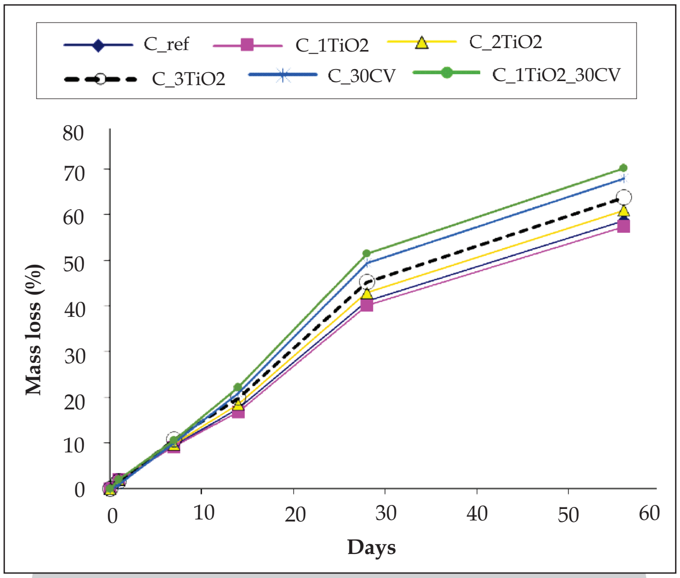

Figure 13. Resistance to sulphuric acid attack

Table 5. Resistance to chloride penetration [27]

$\times 10^{-12}$	Concrete resistance
>15	Low
10-15	Moderate
5-10	High
2,5-5	Very high
<2,5	Ultra high

4.5 Chloride diffusion

The chloride diffusion results are shown in Figure 11. With the exception of the mixture with 3 % nano-TiO_2 content which has a moderate resistance to chloride penetration (Table 5) all the others show a high resistance. The mixture with fly ash and 1% nano-TiO_2 content even shows a very high resistance to chloride penetration. A significant statistical correlation (R^2=0.9) was established between the chloride ion diffusion coefficient and 28 days curing electric resistivity (Figure 12) confirming previous statements [28,29].

4.6 Resistance to sulphuric acid attack

Figure 13 shows the mass loss results after sulphuric acid attack for all the concrete mixtures. Up to 14 days of exposure in acid no noticeable relation between the different mixtures was reported. Nevertheless, after 28 days, a slight difference can be seen. In fact, all the concrete mixtures with fly ash present a higher mass loss. The several mixtures without fly ash show a very similar performance. Still it can be noticed that the 2% and 3% nano-TiO_2 based mixtures have a slightly high mass loss while the 1% nano-TiO_2 mixture shows the best performance. The result of this mixture is in agreement with its compressive strength. It would be expected that the mixture with 1% nano-TiO_2 and 30% fly ash had a lower mass loss because has almost the same compressive strength of the latter which means similar hydration products. The reported behaviour is unaltered even after a 56 days exposure in sulphuric acid.

5. CONCLUSIONS

From the information presented in this paper, the following conclusions can be drawn:

a. A 1% nano-TiO_2 content seems to be an optimal percentage for compressive strength. The increase in the nano-TiO_2 content leads to a decrease in the compressive strength;

b. The results show that the nano-TiO_2 minimizes the strength loss associated to the use of fly ash but only for 28 days curing;

c. In mixtures without fly ash increasing nano-TiO_2 content leads to an increase in water absorption;

d. A significant statistical correlation (R^2=0.98) was obtained between 28 days compressive strength and water absorption by immersion;

e. A significant statistical correlation (R^2=0.98) was obtained between UPS and all compressive strength results;

f. A significant statistical correlation (R^2=0.9) was obtained between chloride ion diffusion coefficient and electric resistivity;

g. Concrete mixtures with fly ash show a higher mass loss after sulphuric acid attack exposure.

h. The mixture with 1% nano-TiO_2 content seems to have the best performance concerning resistance to acid attack

i. The results show that the use of nanoparticles even as low as just 1% leads to a severe increase in the concrete cost. This means that the economic feasibility of nano-TiO_2 based concrete is dependent on the future reduction of the cost of nanoparticles.

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